REMARKS / ARGUMENTS

I. General Remarks

Please consider the application in view of the following remarks. Applicants thank the Examiner for his careful consideration of this application.

II. Disposition of Claims

Claims 15-18, 20-23, 28-30, 47-50, and 55-67 are pending in this application. Claims 1-14, 19, 32-46, and 69-81 were cancelled in a previous response. Claims 24-27, 31, 51-54, and 68 have been withdrawn in response to the Examiner's restriction requirement.

Claims 82 and 83 have been added herein. These additions are supported by the specification as filed.

Claims 15-18, 20-23, 29, 30, 47-50, 56-64, 66, and 67 stand rejected under 35 U.S.C. § 102(b). Claims 15, 23, 28, 47, 50, 55, and 65 stand rejected under 35 U.S.C. § 103(a).

III. Remarks Regarding Rejections of Claims

A. Rejections of Claims Under § 102(b)

Claims 15-18, 20-23, 29, 30, 47-50, 56-64, 66, and 67 stand rejected under 35 U.S.C. § 102(b) as anticipated by U.S. Patent No. 6,387,986 to Moradi-Araghi *et al.* ("*Moradi-Araghi*"). With respect to these rejections, the Office Action states:

> Applicant has amended the bridging agent in independent claims 15 and 47 to be a degradable material. However, dependent claims 22 and 49 define the degradable material (and, thus, the bridging agent) to be, inter alia, (poly)orthoester.
>
> Moradi-Araghi discloses a gel-forming composition comprising a material encapsulated with a degradable first polymer; a second polymer and a liquid (col. 3, line 59-65); a clay may be included as a viscosifier (can be at 0.25 weight percent) and a weighting agent such as calcium carbonate (that can further act as a bridging agent) can also be added (see col. 3, line 66 to col. 4, line 27). The degradable first polymer may be a polyorthoester (see col. 3, lines 12-16) and/or a poly(lactic acid-co-glycolic acid) that can also behave as a plasticizer (col. 3, lines 15-17; claim 3). (See also, instant specification, pages 9 and 10, paragraphs [0026] and [0027] disclosing polylactide polymers as an exemplary plasticizer).
>
> The second polymer can be a carboxymethylcellulose or xanthan gum (may be at 0.01 weight percent) [well known as viscosifiers and fluid loss agents], among others (see col. 5, lines 4-22 and 60-67). The liquid can be water (see col. 6, lines 12-17). The fluids

of Moradi can be used during drilling (see col. 6, lines 60-65). *The capsules of the first polymer can be fairly small (see Example 1 and col. 4)* *and can act as a bridging agent.*

The method step of circulating is implied in the disclosure of use in drilling. *The method steps of* *forming a cake* *and degrading/self-degrading this cake* *are inherent in the materials used here* *when placed downhole.* Note that the capsule can be designed so that gellation is slow and a thin gel may be produced (see col. 4); *this allows for an initial filter cake to form and degrade (at least in part) before a second type of cake is formed*; this second cake may be much thinner and more permeable than the first.

As to claim 29, the definition given at page 6, paragraph 20 of the specification for efficient filter cake is broad enough to include about any situation disclosed by the reference. As to claims 30 and 67, some of the amounts described for degradable material in the reference are within those of claim 30 and 67 (see col. 4 and Example 1).

As to claim 56, fluids within this range are taught (see col. 4-6).

The reference described and contemplates capsules of a degradable polymer that are of small enough size to be useful as a bridging agent. (See, e.g., Example 1 and col. 4) *Larger capsules can be used for this purpose, even if not optimally.*

Thus, the instant claims are anticipated by Moradi-Araghi

(Office Action at 3-4 (emphases added).) In response to Applicants previous remarks, the Office Action states:

> Applicant's arguments regarding the 35 U.S.C. 102 rejection of claims as anticipated by Moradi-Araghi have been fully considered but they are found not persuasive.
>
> Examiner notes that Applicant has presented arguments for the 102 and 103 rejections over Moradi-Araghi. However, in both instances Applicant's arguments regarding Moradi-Araghi not "disclos[ing] a bridging agent comprising a degradable material" are inaccurate. As discussed above, Applicant has limited the bridging agent to be the degradable material. Moradi-Araghi discloses drilling compositions containing poly(orthoester), which are defined in the instant specification and claims to be a degradable material. Consequently, Moradi-Araghi is disclosing the drilling composition to comprise a bridging agent in accordance with Applicant's specification.

(Office Action at 7.) Applicants respectfully disagree with these rejections.

In order to form a basis for a rejection under 35 U.S.C. § 102(b), a prior art reference must disclose each and every element as set forth in the claim. MANUAL OF PATENT EXAMINING PROCEDURE (hereinafter "MPEP") § 2131 (2007). Applicants respectfully submit that *Moradi-Araghi* does not disclose the use of a <u>degradable material bridging agent</u>, as recited in claims 15 and 47, nor does it disclose the steps of <u>forming a self-degrading filter cake that comprises the degradable material bridging agent</u>, and allowing that filter cake to degrade, as recited in claim 15. Applicants hereby incorporate into this response all of their remarks in Section V.A. of their previous response of September 29, 2006 explaining why *Moradi-Araghi* fails to disclose these elements, and further elaborate on those remarks below.

The Office Action assumes that because "dependent claims 22 and 49 define the degradable material (and, thus, the bridging agent) to be, inter alia, (poly)orthoester," those materials in any form will "inherently" act as a bridging agent and form a self-degrading filter cake when placed downhole. (Office Action at 3.) Respectfully, these assumptions are incorrect. Initially, Applicants note that the Manual of Patent Examining Procedure clearly places the burden on the examiner to establish that the crosslinking agents encapsulated with degradable materials disclosed in *Moradi-Araghi* will <u>necessarily</u> act as a bridging agent and form a self-degrading filter cake in a subterranean formation. MPEP at § 2112. The Office Action assumes that they will do so simply because they have the same chemical composition, without citing anything in *Moradi-Araghi* or any other reference to support that assumption. Dependent claims 22 and 49 do recite that the degradable material may comprise one or more different polymers (*e.g.*, poly(orthoesters)), but that recitation does not imply that those polymers in <u>any</u> form will act as a bridging agent or form a self-degrading filter cake. Rather, dependent claims incorporate the additional elements from independent claims 15 and 47, respectively, which require those steps and therefore limit the scope of possible bridging agent materials recited in dependent claims 22 and 49. *Moradi-Araghi* does not disclose or teach any material that will inherently perform these functions. The Office Action hypothesizes from *Moradi-Araghi* that "a thin gel may be produced [which] allows for an initial filter cake to form and degrade (at least in part) before a second type of cake is formed." (Office Action at 4.) This notion of "initial" and "second" filter cakes is similarly unexplained by anything in *Moradi-Araghi* or elsewhere in the art. There is no explanation of how forming a gel will <u>necessarily</u>

lead to the formation of a filter cake, much less a filter cake that comprises the degradable materials recited in *Moradi-Araghi*.

Contrary to the Office Action's assumption, the encapsulated crosslinkers disclosed in *Moradi-Araghi* cannot anticipate Applicants' claims since, even if they comprise the same degradable polymers that Applicants' claimed bridging agents may comprise, those encapsulated crosslinkers are not properly sized to act as bridging agents or to form a self-degrading filter cake within the formation. The Office Action maintains that *Moradi-Araghi* contemplates capsules of a degradable polymer that "can be fairly small" or "of small enough size to be useful as a bridging agent," citing column 4 and Example 1 of *Moradi-Araghi*. (Office Action at 4.) However, neither these portions of *Moradi-Araghi* nor the balance of that reference even mention the size of the particles. *Moradi-Araghi* does mention the molecular weights of certain polymers that may be used to encapsulate the crosslinkers therein (*see Moradi-Araghi* at col. 4, ll. 52-62), but this provides no information whatsoever regarding the particle size of the encapsulated crosslinkers.

Rather than pointing to any particles in *Moradi-Araghi* properly sized to act as bridging agents, the Office Action conjectures that larger capsules taught in *Moradi-Araghi* can be used as bridging agents, "even if not optimally." (Office Action at 4.) To the contrary, it is well known and documented in the art that larger particles randomly chosen without reference to the particle size needed to bridge pore throats between particulates within a subterranean formation will <u>fail</u> to act as a bridging agent, and will <u>fail</u> to form a filter cake. *See, e.g.,* Gatlin, C., and Nemir, C.E., "Some Effects of Size Distribution on Particle Bridging in Lost Circulation and Filtration Tests," Journal of Petroleum Technology at p.576 (June 1961) (Table 1: "no bridge formed and no pressure held" in simulated formations tested with randomly selected sack of medium sized particulates); Mahajan, N.C. and Barron, B.M., "Bridging Particle Size Distribution, A Key Factor in the Designing of Non-Damaging Completion Fluids," Paper SPE 8792, presented at the 4th Symposium on Formation Damage Control, Jan. 28-29, 1980, Bakersfield, Calif. (bridging on formation face "can only be done by proper selection of bridging particle sizes in relation to the formation pore sizes"); U.S. Patent No. 2,815,079 to Goins *et al.*, at col. 2, l. 56 - col. 3, l. 20 (materials having particle size distributions falling outside of a narrow range "were found ... to fail in sealing the larger-size openings even though all of these had particles of the same maximum size"). (Courtesy copies of these references have been

provided for the Examiner's convenience.) Larger particles of degradable materials, like those in *Moradi-Araghi*, would simply sit on top of the pore throats in the subterranean formation, creating new pore spaces between the formation particulates and the particles of degradable materials. Fluid will flow through these new pore throats just as readily as it flows through the original pore throats, and no filter cake will form. Thus, the larger encapsulated crosslinkers in *Moradi-Araghi* will not act as bridging agents, as recited in claims 15 and 47, nor will they form a self-degrading filter cake in subterranean formation, as recited in claim 15.

Moreover, with respect to dependent claim 29, *Moradi-Araghi* cannot anticipate this claim, in addition to the reasons discussed above with respect to claim 15 from which claim 29 depends, because *Moradi-Araghi* does not disclose including an amount of a bridging agent sufficient to form an "efficient filter cake." The Office Action also admits that larger particles in *Moradi-Araghi* may not bridge pore throats and form a filter cake "optimally" (Office Action at 4), acknowledging that any filter cake formed with the particles described in *Moradi-Araghi* may not be an "efficient" one. Indeed, the references cited above indicate that larger particles will not form any filter cake at all, much less an "efficient filter cake" within Applicants' definition.

Therefore, because *Moradi-Araghi* does not disclose these elements recited in claims 15 and 47, as amended herein, *Moradi-Araghi* cannot anticipate these claims, and claims 15 and 47 are allowable over *Moradi-Araghi*. Moreover, since "a claim in dependent form shall be construed to incorporate by reference all the limitations of the claim to which it refers," and since claims 16-18, 20-23, 29, 30, 48-50, 56-64, 66, and 67 depend, either directly or indirectly, from independent claim 15 or 47, these dependent claims are allowable for at least the same reasons, in addition to the reasons discussed with respect to claim 29. *See* 35 U.S.C. § 112 ¶ 4 (2004). Accordingly, Applicants respectfully request the withdrawal of these rejections.

B.   Rejections of Claims Under § 103(a)

Claims 15, 23, 28, 47, 50, 55, and 65 stand rejected under 35 U.S.C. § 103(a) as unpatentable over *Moradi-Araghi* in view of U.S. Patent No. 5,728,652 to Dobson *et al.* ("*Dobson*"), U.S. Patent No. 5,191,931 to Himes *et al.* ("*Himes*"), or U.S. Patent No. 4,531,594 to Cowan ("*Cowan*"). With respect to these rejections, the Office Action states:

> The Moradi-Araghi has been discussed above ... and all the grounds of rejection and arguments therein are incorporated herein in their entirety.

Moradi-Araghi is silent regarding the particle size of the various components of the drilling fluid composition, such as the bridging agent and the degradable polymer.

However, it is well known in the prior art to control the particle size for drilling fluid components, such as bridging agent, a degradable polymer or other fluid-loss control additives dependent on the particular application/formation.

For example, Dobson teaches that bridging agents are routinely sized to have a particular size distribution sufficient to seal off pores, preferably having a particle size of between 5 and 800 microns. (Col. 5, line 51 to col. 6, line 12) Himes teaches that particle sizes of solid materials of a drilling composition are manipulated to bridge formation pores to prevent fluid loss. (Abstract) Cowan discusses design variability of particle size of fluid-loss control additives in drilling fluids to provide enhanced fluid loss prevention, preferably between 74 and 2 microns. (Col. 2, lines 22-26; col. 5, lines 29-43)

Nevertheless, it would have been obvious to one of ordinary skill in the art to control the size of the capsules, and in the course of this routine optimization process, capsules within the range of claim 28 and 55 could have been made which are clearly suitable for use as bridging agents. To the extent that the size of the capsule, such as in Example 1, differs from the size in claims 28 and 55, it would have been obvious to control the size to attain specific degradation times.

Moreover, although Moradi-Araghi may not explicitly disclose adding a plasticizer to the composition (although Moradi-Araghi does disclose adding a polylactide as discussed above), or hydroxyethylcellulose/starch as a fluid loss reducer, it is common in the art to add (modify) a drilling fluid by adding a plasticizer or hydroxyethylcellulose as a viscosifier/fluid loss reducing agent to provide a more efficient method of drilling a subterranean formation. (See, e.g., Dobson, col. 1, line 49 to col. 2, line 25; Himes, col. 1, lines 63 to col. 2, line 12; Cowan, col. 11., lines 8-16)

Therefore, it would have been obvious to a person of ordinary skill in the art to optimize the particle size of the bridging agent/degradable polymer of, and/or add a conventional fluid loss reducer (such as hydroxyethylcellulose or starch) to, the drilling fluid composition disclosed in Moradi-Araghi. One skilled in the art would have been motivated to do so to incorporate the teachings of Dobson, Himes or Cowan and attain a superior drilling fluid having effective bridging of the formation pores and enhanced fluid loss prevention.

Thus, the instant claims are unpatentable over Moradi-Araghi and either Dobson, Himes or Cowan.

(Office Action at 6.) In response to Applicants previous remarks, the Office Action states:

Applicant's arguments regarding the 35 U.S.C. 103 rejection of claims 15, 23, 47, 50 and 65 as unpatentable over Moradi-Araghi have been fully considered but they are not persuasive.

Examiner's discussion of Applicant's arguments concerning Moradi-Araghi of paragraphs #5, 6 and 7 apply equally to the instant rejection and are incorporated herein. Applicant has limited the bridging agent to be, e.g., poly(orthoester). Moradi-Araghi meets this limitation because it discloses the drilling composition to contain poly(orthoester).

(Office Action at 7-8.) Applicants respectfully disagree with these rejections.

In order to form a basis for a rejection under 35 U.S.C. § 103(a), a prior art reference or combination of references must teach or suggest each and every element as set forth in the claim. MPEP at § 2143.03. Moreover, where the rejection relies on a combination of references, there must be some specific teaching or motivation within the knowledge of one of ordinary skill in the art to make that combination. *Id.* at § 2143.01. Where the proposed combination would make the reference being modified "unsatisfactory for its intended purpose," or "change the principle of operation" of that reference, then there is no suggestion or motivation to make the proposed combination. *Id.*

As stated in the Office Action, these § 103(a) rejections depend in part on the Examiner's assertion that *Moradi-Araghi* teaches the same degradable material bridging agent that Applicants' claims recite. However, as discussed in Section III.A above, *Moradi-Araghi* does not teach or suggest a degradable material bridging agent, as recited in claims 15 and 47, as amended herein, nor does it teach or suggest the steps of forming a filter cake that comprises the degradable material bridging agent, and allowing that filter cake to degrade, as recited in claim 15, as amended herein. Nor does *Dobson, Himes,* or *Cowan* supply those missing elements.

Moreover, there is no motivation whatsoever to combine *Moradi-Araghi* with *Dobson, Himes,* or *Cowan* as the Office Action suggests. *Moradi-Araghi* discloses crosslinking agent particulates that are encapsulated with a degradable material to delay gelation of a fluid. *Dobson, Himes,* and *Cowan* describe the use of particulates to bridge pore throats in a subterranean formation. Given these completely different uses, there is no reason that a person

of skill in the art would think that the crosslinking agent particulates of *Moradi-Araghi* might be used as the bridging agents described in *Dobson*, *Himes*, and *Cowan*. Nor is there any teaching or suggestion, in *Moradi-Araghi*, *Dobson*, *Himes*, or *Cowan* that particulates comprising encapsulated crosslinking agents could be used as bridging agents like those disclosed in *Dobson*, *Himes*, and *Cowan*.

The Office Action maintains that it would be obvious to modify the encapsulated crosslinking agents in *Moradi-Araghi* by optimizing the size of those particulates to obtain "specific degradation times," making them suitable for use as bridging agents. (Office Action at 6.) However, there is no reason to believe that the particle sizes to which the crosslinking agents in *Moradi-Araghi* would be optimized in order to obtain those degradation times would coincide with the particle sizes necessary to act as a bridging agent, as Applicants' claims require. Indeed, modifying the encapsulated crosslinking agents in *Moradi-Araghi* to make them small enough to act as a bridging agent may render them unsuitable for the very purpose taught in *Moradi-Araghi* of accomplishing delayed gelation of a fluid, for example, if the degradable encapsulating material is not big enough to encapsulate the crosslinking agent material. *See* MPEP at § 2143.01.

Therefore, the combinations of *Moradi-Araghi* and *Dobson*, *Himes*, or *Cowan* do not teach or suggest all elements of claims 15 and 47, as amended herein, nor would there be any motivation to combine those references in the manner explained in the Office Action. Claims 28 and 55 each depend, either directly or indirectly, from claims 15 and 47, respectively, and thus incorporate all of the elements in claims 15 and 47 that the cited prior art references do not teach. *See* 35 U.S.C. § 112 ¶ 4 (2004). Thus, Applicants respectfully assert that claims 28 and 55 similarly are patentable over those combinations of references, and respectfully request the withdrawal of these rejections.

IV.   New Claims

Finally, Applicants have added new claims 82 and 83 which depends from claims 15 and 47, respectively. In addition to the reasons discussed above in Section III.A. with respect to claims 15 and 47, claims 82 and 83 are allowable over *Moradi-Araghi* because *Moradi-Araghi* does not disclose or teach including the bridging agent in the well drill-in and servicing fluid in an amount sufficient to provide a fluid loss of less than about 15 mL per API Recommended Practice 13. Moreover, it would not be obvious to modify *Moradi-Araghi*, either alone or in

view of *Dobson*, *Himes*, or *Cowan*, to include this amount of the degradable material bridging agent since the cited references do not teach or suggest including the bridging agent in this amount, and, as discussed in Section III.B, there is no motivation or teaching to combine *Moradi-Araghi* with those references.
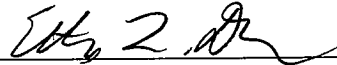
V.   No Waiver

All of Applicants' arguments and amendments are without prejudice or disclaimer. Additionally, Applicants have merely discussed example distinctions from the cited references. Other distinctions may exist, and Applicants reserve the right to discuss these additional distinctions in a later Response or on Appeal, if appropriate. By not responding to additional statements made by the Examiner, Applicants do not acquiesce to the Examiner's additional statements, such as, for example, any statements relating to what the prior art of record discloses and/or what would be obvious to a person of ordinary skill in the art. The example distinctions discussed by Applicants are sufficient to overcome the anticipation and obviousness rejections.

SUMMARY

In light of the above remarks, Applicants respectfully request reconsideration and withdrawal of the outstanding rejections. Applicants further submit that the application is now in condition for allowance, and earnestly solicit timely notice of the same. Should the Examiner have any questions, comments or suggestions in furtherance of the prosecution of this application, the Examiner is invited to contact the attorney of record by telephone, facsimile, or electronic mail.

Applicants believe that there are no fees due in association with this filing. However, should the Commissioner deem that any fees are due, including any fees for extensions of time, the Commissioner is authorized to debit Baker Botts L.L.P.'s Deposit Account No. 02-0383, Order Number 063718.0187.

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Some Effects of Size Distribution on Particle Bridging in Lost Circulation and Filtration Tests

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INTRODUCTION

A common cure of lost circulation is the introduction of granular bridging agents into the mud system. Many materials, such as ground nut shells, are used for this purpose. If the trouble causing void (fracture, fissure, vug, etc.) is not too large, the granular agent forms a "bridge" across or within the opening. Successively smaller particles in the mud stream then accumulate on and/or within this bridge until normal filtration is finally established. The efficiency of many bridging agents has been studied in the laboratory using various slot testers.[1,2]

Many investigators have remarked that a granular bridging material should have the proper particle size distribution. With the exception of the broad range specified in the patent of Goins and Nash,[3] there is apparently no definition as to what this distribution should be.

A bridge may be initiated when several particles of lost circulation material lodge against each other in a fracture or other void. Smaller particles then may bridge the openings between the larger, previously bridged particles. This process continues until the voids become quite small and the problem becomes one of filtration. It would seem logical that an optimum particle size distribution exists, i.e., one containing the proper quantity of properly sized material to fill the successively smaller voids of the bridge.

The filtration characteristics of drilling fluids may be subject to a similar analysis. It has been clearly demonstrated by many workers that the filtration behavior of various muds can be altered by particle size control.[4-8] Slusser, et al,[7] divided filtration into three periods and showed that particle sizes affecting a given period did not necessarily affect other portions of the filtration curve.

An analagous problem is the proper sizing of gravel, liner slots and screen sizes in various sand-exclusion problems. Here the situation is reversed in that a proper "void" size, rather than the size of the bridging agent, is sought. The classic work of Coberly[9] forms the quantitative basis for most sand-exclusion processes. It is interesting to note that, in general, Coberly's work has been overlooked by most drilling-mud researchers.

Other investigators have made various studies of the effect of particle size distribution on drilling-mud properties. As a part of their studies, Fancher and Oliphant reported 30-minute filtration loss of a variety of mixtures of particle sizes.[10] Gates and Bowie noted an effect of particle size distribution on filtration, as well as on other physical properties.[4]

MAXIMUM DENSITY THEORY

A maximum density mixture has been mentioned as the possible solution for both lost circulation and mud filtration problems.[11] Furnas[12] derived mathematically a method for obtaining maximum density of beds of packed solids, having either a given number of component sizes or a continuous gradation of sizes. This work was verified experimentally by Anderegg.[13] For mixtures having a continuous grading of sizes, the interval between sizes is taken as successive screen sizes of interval $\sqrt{2}$. The ratio of the quantity of each size and the next smaller size is given by the equation

$$r = \frac{1}{\phi^{n/m}}$$

where r = the quantity ratio between successive sizes of interval $\sqrt{2}$,

ϕ = the porosity of a bed composed of material of one screen size,

n = one less than the number of component sizes obtained from the ordinate of Fig. 1, and

m = one less than the number of screens used, i.e., the number of size intervals of ratio $\sqrt{2}$.

Fig. 1, number of size intervals of ratio $\sqrt{2}$ taken from Furnas' paper, gives the value of $n + 1$ for any given values of ϕ and K, where K is the ratio of the diameter of the smallest particles to the diameter of the largest particles. Measurement by weight assumes, of course, constant density for all particle sizes. Choosing the quantity of the smallest particle size as a unit weight, the required weights of the successively larger intervals are then r, r^2, r^3, ..., $(r)^m$. Thus, the grading curve of a maximum density mixture represents a geometric progression of ratio r.

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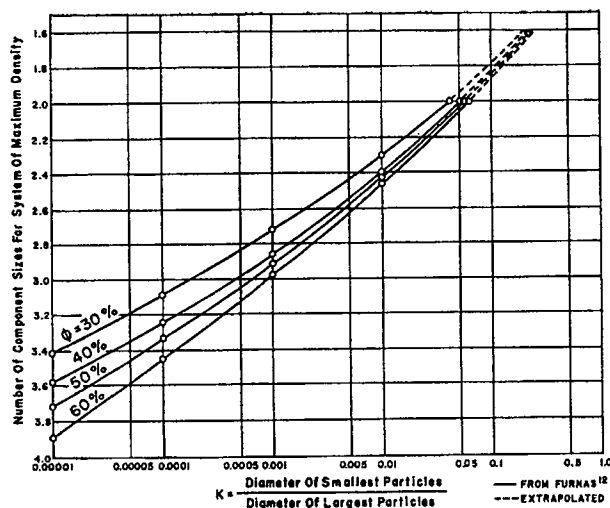
FIG. 1—RELATION BETWEEN SIZE RATIO AND NUMBER OF COMPONENT SIZES FOR SYSTEMS OF MAXIMUM DENSITY.

FIG. 2—PARTICLE SIZE DISTRIBUTION OF RANDOM NUT-SHELL SACK.

The purpose of this study was the preliminary application of the Furnas theory to some particle bridging features of lost circulation and mud filtration.

LOST CIRCULATION TESTS

These tests were conducted with a wedge-shaped slot tester, as described elsewhere.[2] The slot used had an entrance width of 0.50 in., tapering to about 0.06 in. at the exit.

A 50-lb, randomly selected sack of medium-sized crushed walnut shells was screened using United States Standard sieves; the sizes of successive sieves vary by $\sqrt{2}$. The particle size distribution of this sample is shown in Fig. 2. It is realized, of course, that not all sacks will necessarily have this distribution. The maximum density distribution of particle sizes then was calculated by Furnas' method, using $\phi = 0.50$; this distribution curve also appears in Fig. 2.

A nut shell sample having its original particle size distribution was thoroughly mixed into a 16-gal sample of mud in a concentration of 20 lb/bbl. Likewise, a quantity of particles was combined to give the calculated maximum density distribution. This was mixed into an identical portion of mud in the same concentration of 20 lb/bbl.

Each sample was pumped through the tester at about 8 gal/min until bridging occurred, or until the entire sample was expended. A stable bridge occurred when fluid did not pass the slot at 225-psi differential. Results of these tests are shown in Table 1, Runs 1 through 12. It is clear that the maximum density mixture consistently bridged the slot, while that from the original sack did not.

An additional quantity of the largest particles (12 mesh) then was added to the standard sample so that it contained the same amount of the largest size as did the maximum density sample. Runs 13 through 15 were made with this mixture. In Run 13 a stable bridge formed; however, Runs 14 and 15 were unsuccessful. These three tests suggest that, while the quantity of large particles is important in initiating the bridge, a proper distribution of successively smaller sizes supplements the "seal".
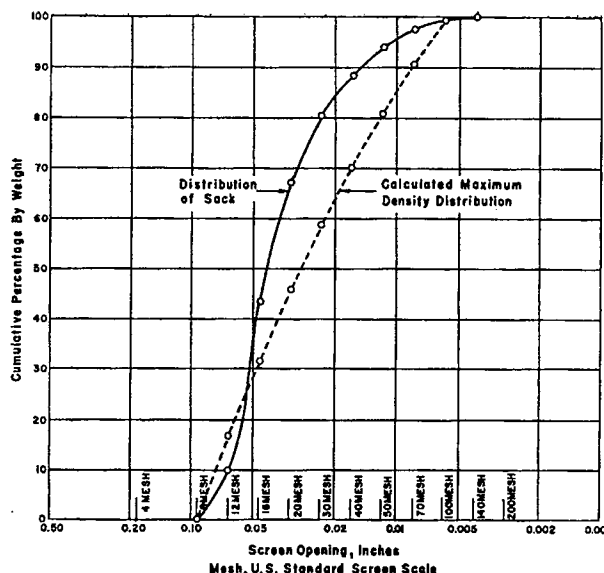
FILTRATION TESTS

Standard API equipment was used for the filtration tests against filter paper. Other tests substituted a 2¾-in. diameter by 2-in.-thick sandstone core for the filter paper. The cores used were quite uniform and had permeabilities of 600 to 800 md.

The fractionated aquagel samples obtained by Jessen and Mungan[14] were used in this investigation. Their nomenclature will be used for each size range as listed in Table 2, taken from their paper. Muds having maximum density, as well as standard particle size distributions were prepared as defined by the following. These will be referred to by number.

MUD

1. 6.1 per cent by weight of particles having the original size distribution of Sizes B through M.

2. 6.1 per cent by weight of the calculated maximum density distribution of Sizes B through M plus 4.23 gm of particle Size M. The extra quantity of Size M was required to furnish viscosity.

3. 4.0 per cent by weight of Size M only.

4. 6.1 per cent by weight of particles having the original distribution of Sizes H through M.

Run No.	Nut Shell Sample	Time for Stable Bridge (sec)	Volume of Mud Pumped Through (gal)	Remarks
	TABLE 1—RESULTS OF WEDGE-SHAPED SLOT TESTS			
1	Random Sack	—	16	No bridge formed and no pressure held on system after test.
2	" "	—	16	"
3	" "	—	16	"
4	" "	—	16	"
5	Maximum Density	43	3	System held 130 psi after test.
6	" "	95	4	"
7	" "	105	5	"
8	" "	10	0	"
9	" "	70	4.5	"
10	" "	70	4.5	"
11	" "	120	8	"
12	" "	135	8	"
13	Added 150 grams of 12-mesh size to random sack sample	45	2	System held 125 psi pressure after test
14	" "	—	16	System held 55 psi pressure after test
15	" "	—	16	15-psi pressure bled-off in 30 sec

TABLE 2—SIZE DISTRIBUTION OF AQUAGEL (AFTER JESSEN AND MUNGAN)[14]

Fraction	Size Range (microns)	Cumulative Per Cent by Weight
B	40	3.76
C	40 — 20	5.56
D	20 — 10	8.41
E	10 — 4	10.81
F	4 — 2	12.91
G	2 — 1	15.91
H	1 — 0.4	20.42
I	0.4 — 0.3	29.42
K	0.3 — 0.15	41.43
L	0.15 — 0.05	62.46
M	0.05	99.99

TABLE 3—PROPERTIES OF MUD SAMPLES

Sample	Plastic Viscosity	Initial Gel Strength (lb/100 ft²)	10-min Gel Strength (lb/100 ft²)
1	25 cp	10	24
2	11.5	5	17
3	6	4	15
4	20	7	26
5	11	2	4

5. 6.1 per cent by weight of the calculated maximum density distribution of Sizes H through M.

After mixing, the flow properties of the mud samples were measured with a Fann V-G meter and are given in Table 3.

A standard filtration test was run and repeated using Sample 1. Using the same procedure, filtration tests were run on Samples 2 and 3. Sample 3 flowed freely through the filter paper without depositing a filter cake. Filtration curves were drawn for Samples 1 and 2 and are shown in Fig. 3. Since the curves become linear, for convenience only the first portion of each curve is shown.

A series of filtration tests then were performed using sandstone cores as the filter septum. Results of these tests are given in Fig. 4.

DISCUSSION OF FILTRATION STUDY

FILTER PRESS TESTS

Two things are immediately apparent from Fig. 3. First, the spurt loss of mud Sample 2 is less than that of Sample 1. A second difference is the steeper slope observed for the filtration of mud Sample 2. Since Sample 2 was composed of the calculated maximum density distribution of particle sizes, except for Size M, this result does not agree with the maximum density idea. This verifies prior work showing that the straight-line portion of filtration is controlled by the highly-hydratable small sizes,[8] which evidently cannot be treated
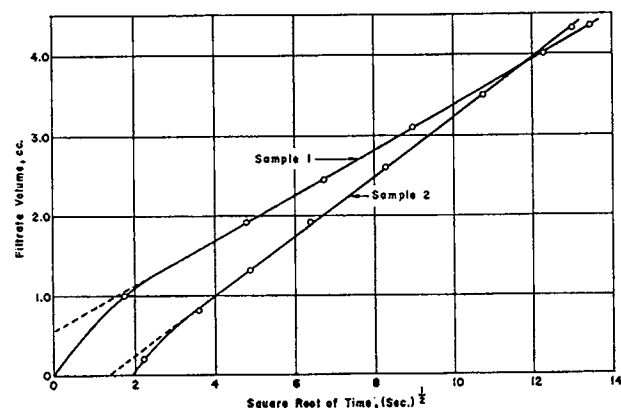

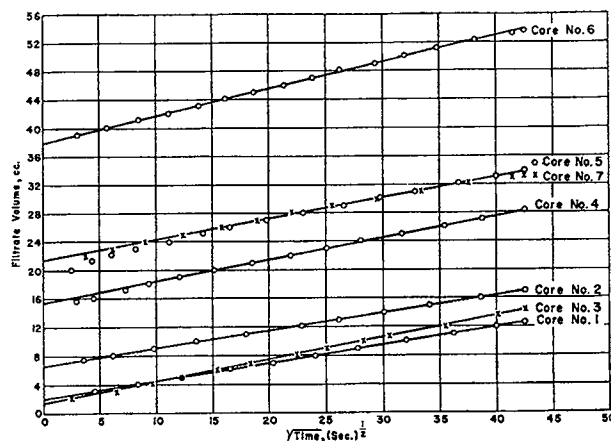FIG. 3—FILTRATION CURVES OF SAMPLES 1 AND 2 ON FILTER PRESS.

FIG. 4—FILTRATION OF MUD THROUGH SANDSTONE CORES.

as inert particles. It appears, however, that the larger sizes (which control the spurt loss) may be treated as inert particles since the maximum density mixture exhibited a reduced spurt loss on filter paper. This confirms the lost circulation results, since lost circulation is little more than a "large-scale spurt".

CORE SAMPLE TESTS

A close similarity between the curve shapes was noted in all of the core sample tests (Fig. 4). The main difference is the spurt loss, which shifts the curves vertically.

In Fig. 4, the spurt loss through Cores 1, 2 and 3 ranges from approximately 2 to 7 cc. These tests were conducted with Muds 1 and 2, containing the full range of particle sizes from B to M. Mud 2 (maximum density sample) was used on Core No. 3, and it is seen that filtration through this core had a slightly smaller spurt loss than that of mud Sample 1 through Cores 1 and 2, but had a steeper slope and, hence, a larger 30-minute water loss. This is in qualitative agreement with the previously discussed results from the filter-press experiments; however, the spurt loss difference is quite small. Slight variations in pore entry sizes of the cores may obscure the effect. The filtration tests of Samples 4 and 5 against the cores show a high spurt loss, ranging from about 15 to 38 cc. This was from two to six times as high as the spurt loss obtained from the mud samples containing the larger particles. Note from Fig. 4, however, that all the curves have substantially the same slope once bridging occurs. There was no significant difference between the filtration characteristics of mud Samples 4 and 5. Therefore, the increased spurt loss was essentially due to the deletion of the large particle sizes with a negligible contribution from the maximum density effect.

Permeability measurements were made on the core samples before and after the tests. In all cases the bulk (75 per cent or more) of formation damage occurred in the first 0.25 cm, as expected. In general, for a given particle size range, the damage depth was less for the maximum density mixture. The data were, however, scattered and inconclusive.[15]

SPECULATIONS

In rotary drilling, the selection of a drilling fluid often involves a considerable compromise between hole conditioning and penetration rate. Much penetration-

rate reduction has been attributed to the "chip hold-down" forces which accompany low-water-loss, high-solid-content, high-viscosity, etc., muds, even in "impermeable rocks". These forces are a consequence of a high pressure difference, or more correctly, a high pressure gradient across the hole bottom, i.e., the element being drilled.[10-18] It also is well known that beneath bit filtration is something quite different from that measured by the API test.[19] Because of the continual bit action beneath bit, filtration is better described by some early portion of the filtration curve. A high spurt mud will clearly result in a reduced pressure gradient across bottom, thereby facilitating cutting removal. The use of clear water is an example.

Deletion of "large" particles from colloidal drilling muds results in high spurt muds. Once bridging occurs, however, the behavior is apparently normal. Thus, it appears possible to construct a mud which can satisfy hole-conditioning requirements with less sacrifice of drilling rate. While this is somewhat conjectural, the principle seems sound, although it is realized that continual maintenance of such a mud may be difficult.

Many other possibilities exist for application of the Furnas theory. The selection of aggregate for gravel packs and, possibly, the sizing of particles for filtration control in other wellbore fluids are typical examples. The Furnas approach can even be applied to the packing of unconsolidated cores for laboratory purposes.

Because of the limited testing performed, the following conclusions are necessarily restricted to the few systems studied. The results are, however, sufficiently promising to encourage further work.

CONCLUSIONS

1. A maximum density distribution of nut-shell particle sizes is more efficient in bridging and sealing a wedge-shaped slot than the distribution found in a randomly selected sack.

2. A maximum density distribution of the "large" particles in commercial bentonite reduces the initial surge or "spurt loss" of a bentonite mud in the standard filter press test.

3. The maximum density theory, which is based on inert particles, apparently does not hold for the linear portion of the filtration curve of a bentonite mud.

ACKNOWLEDGMENT

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RIDGING PARTICLE SIZE DISTRIBUTION: A KEY FACTOR
N THE DESIGNING OF NON-DAMAGING COMPLETION FLUIDS

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ABSTRACT

Great care must be exercised in the formulation
and use of completion and workover fluids; otherwise
extreme damage can be done by "non-damaging" fluids.
Excessive loss of brines, polymers, or bridging solids
to a producing strata can result in irreversible for-
mation damage. Rapid formation of an effective filter
cake on the formation face with the resultant low
filtration rate and minimum solids penetration can sig-
nificantly reduce the potential for formation damage.

Laboratory data indicates that low filtration
rates achieved with the formation of a thin, competent
filter film composed of properly sized bridging par-
ticles and hydrocolloid polymers will most effectively
prevent formation damage. The deposited filter film
acts as a downhole filter, screening out unwanted
solids from the small volume of filtrate actually
entering the formation.

INTRODUCTION

Fluids and solids invading the production zone
cause formation damge. The most successful method
of minimizing damage is to establish a low permea-
bility filter film at the wellbore face as rapidly
as possible. The filter film should be easily broken
down when the well is put back cn production. The
importance of using non-damaging completion and work-
over fluids has long been recognized and close atten-
tion is being given to the problem in the field
operations. However, some factors in designing and
applying these fluids are often overlooked; the result
is a "non-damaging fluid" causing formation damage.
One of the critical factors in designing non-damaging
fluids is obtaining surface bridging on the formation
face with minimum indepth solids penetration. This
can only be done by proper selection of bridging
particle sizes in relation to the formation pore sizes.
The use of acid degradable additives in a wellbore
fluid does not assure protection for a producing zone.
Such additives merely make removing formation damage
more practical or possible.

The most commonly used completion fluid additives
are the hydroxyethyl cellulose (HEC) polymer and
sized calcium carbonate bridging solids. The objective
of this study was to investigate the interaction of
calcium carbonate particle size distribution and hydro-
colloid polymers. Previous studies have related
surface bridging and particle size distribution in
conventional drilling fluids. [3,5,9]

Brines filtered through 2-5 micron filters are
used extensively in completion and workover operations.
Filtered brines are best used where formation permea-
bility is not significantly affected by liquid in-
vasion or where fluid loss to the formation is minimal.
However, field tests have indicated the need for main-
taining exceptionally clean equipment on the rig to
prevent the filtered fluid from picking up rust, scale,
or other debris and carrying it downhole. [6,7]

The addition of properly formulated fluid loss
control additives to the brine protects the formation
from both solid and liquid invasion. The formation
of a thin filter film on the face of the wellbore will
essentially filter out all of the solids down to and
including colloidal size particles. Further fluid loss
is significantly reduced. The filter film then is a
more effective method of solids removal than surface
filters, but success with this approach depends upon
the ability of the fluid to quickly establish a filter
film on the formation surface.

POLYMER ADDITIVES

Certain polymers will raise the viscosity and
improve the carrying capacity of water. (The polymers
most commonly used in the oil fields are long chain
molecules which hydrate in water -- hydrocolloids.)
Other polymers will lower fluid loss when used with
bridging solids. Polymers are in the colloidal size
range and help in the filtration control, once the
primary bridge has been established by the coarser
inert bridging particles.

Core test data and field results indicate that
HEC is the least damaging of the viscosifying

olymers most often used in the oil field.[8] The re-
ults in Table 1 were obtained by digesting 10 g of
he polymer in 700 cm³ of 15% hydrochloric acid while
tirring the sample at 46 K for two hours. The solu-
ion was filtered and the residue washed, dried, and
eighed. HEC has a significantly less acid insoluble
esidue, but if lost in excessive amounts even HEC
an cause damage. Any polymer should be used in the
inimum concentration that will adequately do the job.
t is best to minimize polymer loss to the formation
y having an effective filter film.

Besides being the least damaging polymer, HEC has
ther desirable fluid properties. Being a nonionic
olymer, it will hydrate in all the common brines up
o their saturation point including calcium bromide
rine. This gives the polymer fluid system a wide
ensity range without having to resort to a high
olids weighted system. Further, HEC polymer does
ot develop a significant gel structure under static
onditions, which greatly facilitates the settling of
oreign solids under static surface conditions and
elps to maintain a clean system.

FLUID LOSS CONTROL ADDITIVES

Effective fluid loss control is achieved using a
ombination of inert bridging solids and hydrocolloids.
he formation pores are first bridged by the coarser
ridging particles followed by the finer particles
uccessively filling up the interstitial spaces be-
ween the coarser particles to form a tightly packed
ayer. This stage of filter film build up is accom-
anied by a spurt type fluid loss during which whole
luid is lost into the formation.[5] The fine inter-
article voids in this low permeability layer are then
atisfactorily bridged by the hydrocolloids. At this
oint, filtration, which is characterized by slow,
niform flow of fluid through the filter cake, com-
ences. During filtration, all solids in the fluid
edium are filtered on the surface of the filter film
ith only clear liquid invading the formation.

The volume of spurt loss is controlled by the size
istribution and concentration of the inert bridging
olids. The extent of bridging solid invasion into
he formation pores depends upon the particle size
ange in relation to the pore size distribution. The
aximum density distribution of particle sizes can be
alculated by the method proposed by Furnas. Previous
tudies have looked at solid particle size distri-
ution versus pore size distribution without hydro-
olloids present. The conclusion was that the median
article size should be a third of the median pore
ize.[9] A recent paper, which looked at calcium car-
onates and HEC, concluded that the one third rule
ight not hold.[12] Calcium carbonate bridging particles
re most widely used in workover and completion
operations because of their blocky shape, acid solu-
ility, and availability in a variable size range.
he presence of hydrocolloids could affect the re-
uired ratio of solid particle size distribution to
ore size distribution for effective bridging. Static
nd dynamic filtration tests indicate starch to be a
onsistently effective colloid for fluid loss con-
rol.[11] Another widely used fluid loss control agent
s calcium lignosulfonate. This paper looks at the
wo fluid loss control agents when used with HEC and
alcium carbonate.

FILTER CAKE CLEANUP MECHANISM

The filter film formed using HEC polymer and
calcium carbonate bridging particles is quite thin
as compared to the conventional drilling mud filter
cake. The thickness of a filter cake depends on the
volume of fluid loss and on the solids concentration
in the fluid. A properly formulated polymer based
calcium carbonate system will form a filter film less
than 0.8 mm (1/32 of an inch) in thickness. Such
a film has no inherent structural strength. The
filter film is supported by the formation wall so long
as overbalanced conditions are maintained. When the
well is swabbed or returned to production, the flow of
fluids into the wellbore will mechanically break down
the filter cake by flushing the carbonate particles
from the pores. This cleanup mechanism is possible
only when solids are stopped at the face of the well-
bore. Further, the HEC polymer, which acts as mor-
tar between the carbonate bricks, degrades with time
and lowered pH. The filter film can be acidized as
a remedial step. Care must be exercised in acidi-
zation since the acid will channel to the more per-
meable sections. A laboratory demonstration of
channeling is shown in the following test using a
double Hassler sleeve loaded with two Berea Sandstone
cores. The cell was loaded with a high and a low
permeability core and damaged with a HEC calcium car-
bonate fluid. After that the cores were acidized with
15% hydrochloric acid and the injectivity into the
cores measured. The results are shown in Table 2.
The filter cake on the high permeability core was
broken down first. As a result, no acid was injected
into the low permeability core. Also, acidizing can
have an adverse effect on some formations. There-
fore, the primary cleanup mechanism should be the
natural break down of the polymer and mechanical
flushing of the bridging solids. This will occur
only when there is minimal indepth solids penetration.

EXPERIMENTAL PROCEDURE

The experiment involved core damage tests con-
ducted under static conditions on high permeability
Berea Sandstone cores (500 to 800 md). The first
fluid samples tested were 5% salt brine containing
2.9 kg/m³ (1 ppb) HEC polymer and 10.0 kg/m³ (3.5 ppb)
of calcium carbonate bridging particles. The second
fluids tested were the base fluid above plus various
amounts of starch and calcium lignosulfonate. All
of the polymer additives degrade in a neutral or acid
medium. The cores had been previously evacuated and
saturated with 13% salt brine. The core permeability
was measured using Soltrol (parafinic distillate) as
the flowing phase under ambient conditions at 103 kPa
(15 psi). The initial core permeability was de-
termined with flow in one direction. Damage with the
test fluid was with flow in the opposite direction at
2,068 kPa (300 psi). The fluid loss was measured.
The fluid loss was not allowed to exceed one pore
volume of the core (less than 7 cm³). If higher
volumes were lost, the cleanup results became incon-
sistent. Since the purpose of these tests was to
evaluate formation damage as a result of particle in-
vasion, maintaining a constant fluid loss ensured
that an equal amount of solids from each sample in-
vaded the core. The calcium carbonate bridging
particles were ground marble. Return permeability
was determined with flow in the original direction.
Then slices of the core were removed from the damaged

end. After each slice, the permeability of the remaining core was measured. In this manner, the depth of damage for each core was determined.

INTERPRETATION OF TEST RESULTS

In examining the core damage test data, two properties of the fluid are being evaluated. First, the ability of the test fluid to rapidly form a filter film that maintains a low fluid loss and second, the ease of removal of that film.

The core damage section of the test was stopped before losing more than one pore volume (7 cm^3) of the test fluid to the core. In several instances this meant stopping in less than one minute.

Table 3 indicates about 90% cleanup for HEC polymer. The addition of starch improves the fluid loss but significantly increases formation damage.

Table 4 presents the return permeability for HEC with various size distributions of calcium carbonate bridging particles. Figure 1 shows a plot of the size distribution of the several calcium carbonates used in the tests. For CaCO3-C, CaCO3-D, and CaCO3-E the return permeability did not change much with depth of the core. Product CaCO3-D exhibited better fluid loss control than CaCO3-C or CaCO3-E, but the damage was fairly deep (0.64 cm). The particles size distribution of product CaCO3-D is fairly evenly spread out between 3 to 25 microns with a median of 7.5 microns. The median for CaCO3-C is 9.6 microns and the median for CaCO3-E is 0.81 microns. Product CaCO3-A, which has a similar size range but has a median size of 17 microns, had a high spurt loss. The depth of invasion was much less and the return permeability was better. Product CaCO3-B (median size 41 microns) is too coarse to provide fluid loss control. Consequently the depth of invasion was higher than CaCO3-A, but less than CaCO3-C, CaCO3-D, and CaCO3-E. The best shut off was obtained with CaCO3-D, but the least indepth invasion was obtained with CaCO3-A.

Table 5 presents the return permeability results when starch or calcium lignosulfonate is added to the HEC-calcium carbonate mixtures. There was a significant improvement in fluid loss control, depth of invasion, and return permeability for all calcium carbonate samples. Product CaCO3-D with starch in Sample b had an effective shut off but the polymer did cause some damage deep in the core. By adding calcium lignosulfonate, as in Sample c, only surface damage occured. The best performance for fluid loss control, depth of invasion, and return permeability was with Samples c and e. Figure 3 is a plot of return permeability versus depth of the core.

Figure 2 is a plot of the typical pore size distribution for the high permeability Berea Sandstone cores used. The median pore size is 33 microns.

The testwork done here is limited in scope and additional work will have to be done to confirm the above observations. Such work needs to be conducted under dynamic conditions to simulate field conditions. The concentration of the bridging particles is also a critical factor and additional work is needed in that area.

CONCLUSION

The following are the conclusions drawn from the return permeability test work done.

1) A fluid that contains appropriate fluid loss control additives as well as hydroxyethyl cellulose and calcium carbonate bridging solids gives better fluid loss control with better return permeabilities than a fluid that only contains HEC and calcium carbonates.

2) The use of fluid loss control polymers with HEC and calcium carbonates bridging particles broadens the range of acceptable particle size distributions that will be non-damaging.

3) Depth of solids penetration for a properly designed fluid will be shallow.

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Table 1
Acid* Solubility of Polymers

Polymer Type	Percent Residue
Xanthan Gum**	3.94
Guar Gum	5.20
Hydroxypropyl Guar (HPG)	0.82
Hydroxyethyl Cellulose (HEC)	0.08

* 15% hydrochloric acid
** Xanthan Gum had to be cooked overnight to get sufficient breakdown to obtain filtrate.

Table 2
Acidization Data on Low and High Permeability Cores

	High Permeability Core	Low Permeability Core
Initial Permeability, md	801	167
Fluid loss at 2068.4 kPa for 2 h, cm^3	18	12
15% HCl injection at 344.7 kPa for 10 min., cm^3	12	0

Table 3
Core Damage Test Data — Polymers

Fluid Sample‡ (Concentrations in kg/m^3)	Initial Permeability** (md)	Fluid Loss (cm^3)	% of Original Permeability (Total Length of Core Cut, cm)		
a 2.9 HEC	607	5.6*	94.9 (0)	88.9 (0.828)	93.4 (1.523)
b 2.9 HEC	713	5.6*	94.3 (0)	98.9 (0.506)	
c 2.9 HEC + 2.9 Starch	539	5.8	37.7 (0)	47.1 (0.102)	83.1 (0.855)

‡ Concentrations of additives are rounded off to the nearest tenth.
* Fluid loss in less than one minute.
** Permeability values are rounded off to the nearest whole number.

Table 4
Core Damage Test Data — HEC + Carbonates

Fluid Sample‡ (Concentrations in kg/m³)	Initial Permeability** (md)	Fluid Loss (cm³)	% of Original Permeability (Total Length of Core Cut, cm)			
a 2.9 HEC + 10.0 CaCO₃ — E†	576	5.2*	63.5 (0)	74.3 (0.160)	77.1 (0.704)	77.4 (2.044)
b 2.9 HEC + 10.0 CaCO₃ — C†	715	11.1*	61.5 (0)	69.2 (0.142)	70.5 (0.865)	
c 2.9 HEC + 10.0 CaCO₃ — C†	496	5.6*	81.8 (0)	90.9 (0.169)	74.4 (1.048)	
d 2.9 HEC + 10.0 CaCO₃ — D†	694	5.6	56.3 (0)	65.7 (0.084)	62.8 (0.641)	
e 2.9 HEC + 10.0 CaCO₃ — D†	660	5.2	69.1 (0)	70.2 (0.124)	73.6 (0.630)	
f 2.9 HEC + 10.0 CaCO₃ — A†	585	6.2*	87.8 (0)	89.2 (0.094)	101.0 (0.858)	
g 2.9 HEC + 10.0 CaCO₃ — B†	486	5.4*	63.5 (0)	75.0 (0.191)	93.2 (1.201)	93.4 (2.533)

‡ Concentrations of additives are rounded off to the nearest tenth.
* Fluid loss in less than one minute.
** Permeability values are rounded off to the nearest whole number.
† CaCO₃ — A, B, C, D, E — are all calcium carbonates but with different particle sizes and distribution.

Table 5
Core Damage Test Data — HEC + Carbonates + Starch/Lignosulfonate

Fluid Sample‡ (Concentrations in kg/m³)	Initial Permeability** (md)	Fluid Loss (cm³)	% of Original Permeability (Total Length of Core Cut, cm)		
a 2.9 HEC + 8.6 CaCO₃ — D† + 1.4 Ca Lignosulfonate	696	5.4*	88.8 (0)	96.2 (0.140)	
b 2.9 HEC + 8.6 CaCO₃ — D† + 1.4 Starch	558	2.3	80.1 (0)	88.9 (0.154)	85.7 (0.972)
c 2.9 HEC + 7.1 CaCO₃ — D† + 1.4 Starch + 1.4 Ca Lignosulfonate	551	5.2	90.0 (0)	97.0 (0.067)	
d 2.9 HEC + 7.1 CaCO₃ — C† + 1.4 Starch + 1.4 Ca Lignosulfonate	936	6.2*	69.0 (0)	83.5 (0.168)	
e 2.9 HEC + 7.1 CaCO₃ — A† + 1.4 Starch + 1.4 Ca Lignosulfonate	642	4.8	91.4 (0)	99.7 (0.126)	
f 2.9 HEC + 7.1 CaCO₃ — B† + 1.4 Starch + 1.4 Ca Lignosulfonate	809	5.6*	92.4 (0)	98.6 (0.134)	

‡ Concentrations of additives are rounded off to the nearest tenth.
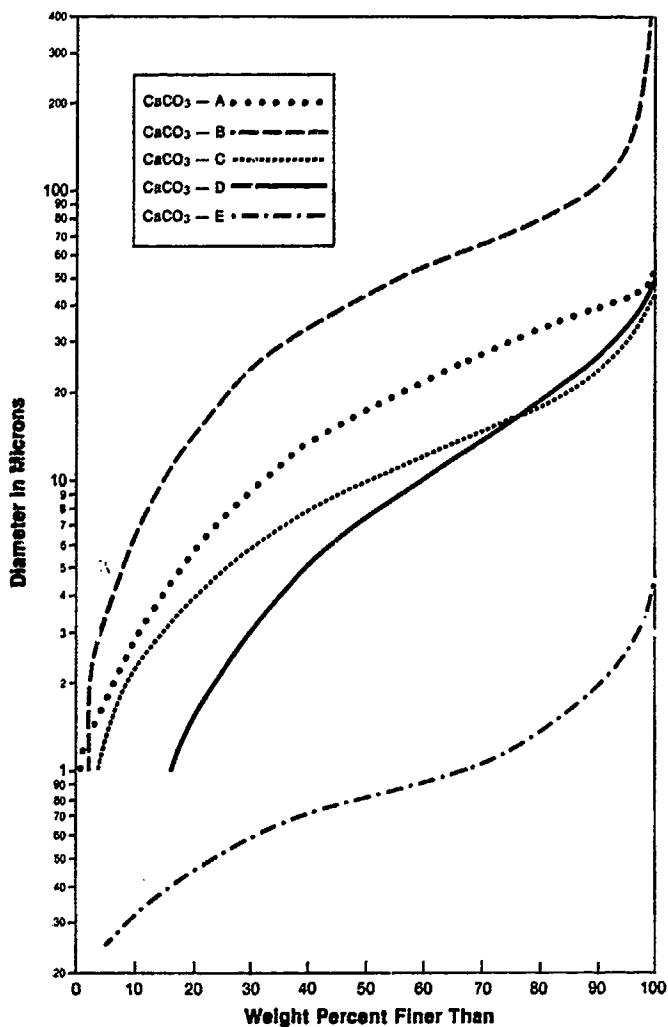* Fluid loss in less than one minute.

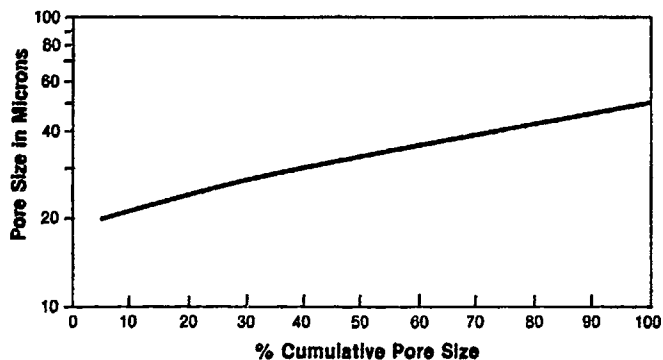Fig. 1 - Typical particle size distribution curves.

Fig. 2 - Pore size distribution of high permeability core.

1 2.9 HEC

2 2.9 HEC + 10.0 CaCO₃ — E

3 2.9 HEC + 10.0 CaCO₃ — C

4 2.9 HEC + 10.0 CaCO₃ — D

5 2.9 HEC + 10.0 CaCO₃ — A

6 2.9 HEC + 10.0 CaCO₃ — B

7 2.9 HEC + 8.6 CaCO₃ — D
   + 1.4 Ca Lignosulfonate

8 2.9 HEC + 8.6 CaCO₃ — D
   + 1.4 Starch

9 2.9 HEC + 7.1 CaCO₃ — D
   + 1.4 Starch
   + 1.4 Ca Lignosulfonate

10 2.9 HEC + 7.1 CaCO₃ — C
    + 1.4 Starch
    + 1.4 Ca Lignosulfonate

11 2.9 HEC + 7.1 CaCO₃ — A
    + 1.4 Starch
    + 1.4 Ca Lignosulfonate

12 2.9 HEC + 7.1 CaCO₃ — B
    + 1.4 Starch
    + 1.4 Ca Lignosulfonate
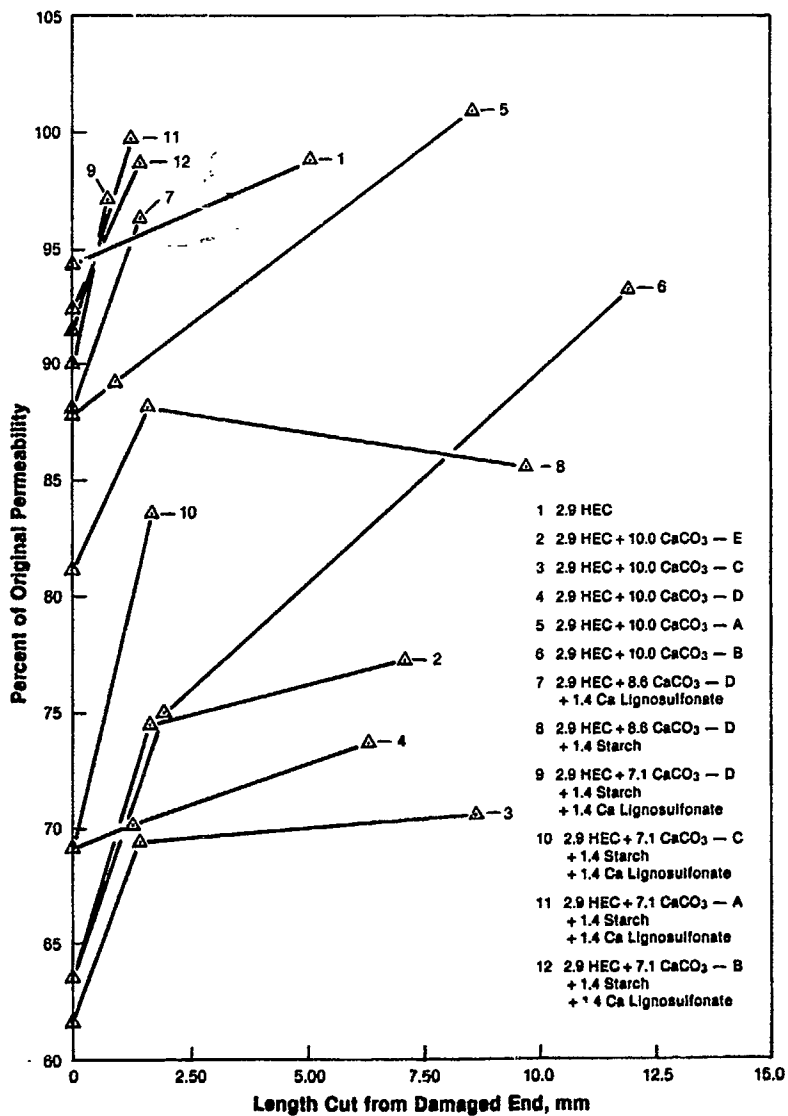
Fig. 3 - Percent of orginal permeability vs damage depth.